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OF POLYMER SOLUTIONS**



TECHNICAL REPORT

By

Ralph L. LeMar

19960405 021

May 1967

**U. S. ARMY WEAPONS COMMAND
ROCK ISLAND ARSENAL
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DA #1CO24401A108

AMS Code 5025.11.802

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FACTORS AFFECTING SONIC DEGRADATION OF POLYMER SOLUTIONS

RALPH L. LEMAR

Rock Island Arsenal, Rock Island, Ill.

Linear polymers dissolved in mineral oil were examined for their lability to shear degradation utilizing a sonic oscillator. The tests were performed on several polyisobutylenes and polymethacrylates similar to those used to formulate hydraulic fluids with improved viscosity-temperature slope properties. The shear lability of these additives varied directly with their viscosity-temperature slope improver power. Slope improver power also was lost during sonic degradation, its extent depending upon the viscosity-temperature slope of the unsheared solutions. The relative shear lability of the polymer solutions was affected by the severity level employed in the sonic tests. Polymethacrylate solutions could be degraded further than polyisobutylene solutions when additives of equivalent oil thickening power were used; however, none were sheared to the viscosity level of the base oil.

MODERN hydraulic and lubricating oil formulations frequently employ as additives linear polymers which cause the formulations to show a low rate of change of viscosity with change in temperature—e.g., 10W-30 motor oils. These polymers can also be made to lower the pour point, improve lubricity, and/or act as detergents.

Polymeric additives in oils are subject to a type of degradation rarely observed for other types of oil additives or pure mineral oils. They can lose part of their ability to increase the base oil viscosity when the polymer molecule is ruptured, which results in shorter polymer chains. Since the oil-thickening effect of the polymeric additive is a function of the chain length, any breakdown or shearing of the molecular chains reduces the viscosity of the polymer-oil blends. Such permanent shear can occur under various types of high energy input to the fluid, such as turbulent flow, high temperatures, sonic treatment, and gamma irradiation. Mechanical components, such as hydraulic pumps, relief valves, and gears, that induce high fluid shear rates and turbulent flow can cause permanent shear.

The potential seriousness of this degradation can be shown by consideration of typical wide-temperature-range fluids such as hydraulic oils described by Military Specifications MIL-H-5606B (20) and MIL-H-13866A (27). These fluids

owe 70 to 90% of their kinematic viscosity to a polymeric additive present in the formulations. Polymer scission or shear degrades these fluids to a much lower viscosity level.

Previous laboratory evaluations of shear stability (resistance of polymer additive to scission) have been generally made via pump loops or fuel injectors, wherein the fluid is recirculated through an orifice with a concomitant large pressure drop (10, 11). These methods are generally costly, slow, and/or subject to frequent breakdown.

A faster and more convenient sonic apparatus for replacement of mechanical shear methods has received wide attention. Scission or shear of polymers in solution is caused by sonically induced cavitation within the fluid sample. This phenomenon is physically similar to turbulence that occurs in oils under high flow rates in mechanical systems. When these bubbles or cavities collapse quickly, pressures and shear forces, theoretically very large, are produced.

In some of the early studies of this apparatus, Jellinek and White (8) found that long-chain molecules in solution are permanently sheared to an intermediate chain length independent of original length, but affected by sonic power level, solvent type, and polymer concentration. Saini and Ostacoli (15) confirmed these conclusions. Prudhomme and Grabar (14) determined that cavitation is important in ultrasonic

depolymerization. In an investigation of the ASTM sonic shear test, Lawson (9) found the shearing of polymethacrylate additives to be much more rapid than in pump test systems but the results were comparable. Gironda, Essing, and Rubin (6) made similar comparisons on military hydraulic oils and related sonic exposure time to mechanical test time. Van Horne (22) found resistance of acrylate and methacrylate polymers to ultrasonic treatment to be determined primarily by molecular weight. Nejak and Dzuna (12) and Trop (19) compared polymer-thickened oils in mechanical and sonic tests and correlated the results. Thomas (18) indicated that the rate of shear was proportional to the product of polymer chain length and diameter. Foster and Mueller (5) found the side-chain length of acrylate and methacrylate polymer additives to affect their sonic shear stability and also found satisfactory correlation between mechanical and sonic test results. Neunherz (13) compared polymethacrylate and polyisobutylene solutions; polyisobutylenes showed superior stability in regard to both permanent viscosity and change in viscosity-temperature properties. Other recent evaluations of the sonic shear method have been unfavorable. LeMar and Bootzin (11) reported on cooperative tests among four laboratories wherein hydraulic fluids were sheared in three sonic oscillators and two different mechanical methods. Discrepancies found between sonic and pump test results were attributed to differences in response of polyisobutylene and polymethacrylate additives to sonic and mechanical treatment. Vick and Goodson (23) also described poor agreement between the two test types, using three chemically different polymers.

Despite correlation problems, the relative speed and simplicity of the sonic method had established its value. Consequently additional studies were performed to clarify effects of polymer type, molecular weight, and solution properties on sonic degradation of polymers.

Experimental

The polymers and the base oil used are listed and described in Table I. The polymers were representative of two chemical types used commercially for improvement of the viscosity-temperature (V-T) properties of oils. "Low" molecular weight versions of the two types possessed nearly equal oil-thickening power at 100° F., as did the "high" molecular weight versions of each polymer type. This is illustrated by the relative viscosity data shown in Table I.

Polyisobutylene molecular weights were determined by intrinsic viscosity techniques as described by Billmeyer (4). Similarly determined data for the polymethacrylates were supplied by Stringer (17).

Five and 15 weight % solutions of each polymer were prepared. Prior to use, the polymer solutions were slowly filtered through 0.45- and 0.20-micron pore size membrane filters in series using a pressure filtration funnel. Particulate contamination was thereby reduced to a low level, in the event that this factor might introduce a variable effect on fluid cavitation during sonic treatment.

Kinematic viscosities of the base oil and polymer blends were determined at 100° and 210° F. by ASTM Method D 445-61 (7). ASTM slopes were determined from the fluids' kinematic viscosities at 100° and 210° F. The ASTM slope is defined as:

$$\text{ASTM slope} = -\text{tangent } A = Y \text{ cm./}X \text{ cm.}$$

where A = angle between viscosity-temperature line and the lines of constant viscosity. It is calculated from the slope of the line obtained when viscosity (Y axis) is plotted *vs.* tempera-

Table I. Description of Polymer and Base Oils

Material	Chemical Name	Molecular Weight	Relative Viscosity at 100° F.	
			5 wt. %	15 wt. %
A-L	Poly(<i>n</i> -octyl) methacrylate	92,000	1.71	4.85
B-L	Polyisobutylene	2,600	1.58	3.90
A-H	Poly(<i>n</i> -octyl) methacrylate	342,000	4.31	25.7
B-H	Polyisobutylene	44,000	3.67	24.4
Base oil	Mineral oil		1	1

ture (X axis) on ASTM chart D341. This value varies directly with a fluid's V-T properties—i.e., an increase in ASTM slope corresponds with an increased rate of change of fluid viscosity with temperature.

In this work, ASTM slopes were used in preference to the more common viscosity index (ASTM Method D 2270) (2). In this regard, Zuidema (24) stated that the ASTM slope is the most fundamental among the three commonly used systems that also include the viscosity index and viscosity pole height. Hatton (7) stated that the ASTM slope shows no irregularities over a wide range of V-T properties. Because the ASTM slope is used in this study, the term, "V-T slope improver" is used in lieu of the more common "V. I. improver" in subsequent discussions on this property.

The sonic treatment employed a Raytheon Model DF101 250-watt sonic oscillator operated at 10-kc. frequency, assembled according to the proposed ASTM method (3). The oscillator coil polarizing current was maintained at 7.3 ± 0.1 amperes. In this apparatus the amount of polymer shear was increased by decreasing the sample volume, increasing the applied power which was proportional to the amperage output of the oscillator, and increasing the time of exposure. The amount of polymer sheared per unit time decreased gradually and eventually approached zero as test time was extended. To determine effects of these parameters, three test conditions were selected:

Sonic Test Parameters

Test No.	Input Power, Amperes \pm 0.025	Sample Volume, Ml. \pm 0.5
1 (most severe)	1.5	15
2	0.5	15
3 (least severe)	0.5	30

The polymer solutions were exposed for time periods that ranged from 2.5 to 320 minutes. Polymer shear was measured as changes in the solutions' kinematic viscosity at 100° F.

Results and Discussion

The polymer solutions' rate of viscosity change per minute of sonic treatment approached zero after 80 to 160 minutes of exposure. Table II shows viscosity and ASTM slope data for the polymer solutions before and after 160 and 320 minutes of sonic treatment, and effect of sonic treatment on the loss of polymer contribution to viscosity. This value was used to compare shear stability and is defined as follows:

Fraction lost of polymer contribution

$$\text{to solution viscosity} = \frac{n_1 - n}{n_1 - n_0}$$

where n_1 = solution viscosity

n = solution viscosity after shear

n_0 = viscosity of base oil

Table II. Effect of Sonic Treatment on Polymethacrylate and Polyisobutylene Solutions

Material ^a	Concn., Wt. %	Sonic Treatment			Viscosity, Cs.		ASTM	Polymer ^b
		Time, min.	Power, amp.	Sample volume, ml.	100° F.		Slope 100— 210° F.	Contribution Lost
					100° F.	210° F.		
Base oil	100	3.62	1.33	0.86	...
A-H	5	0	15.61	6.22	0.42	...
		160	0.5	30	6.70	2.48	0.62	0.743
		160	0.5	15	6.22	2.27	0.66	0.783
		320	0.5	15	6.18	2.26	0.66	0.786
		320	1.5	15	5.40	1.95	0.71	0.852
A-H	15	0	93.08	31.52	0.30	...
		160	0.5	30	28.17	8.95	0.45	0.726
		160	0.5	15	23.16	7.54	0.45	0.782
		320	0.5	15	22.13	7.31	0.46	0.793
		160	1.5	15	14.04	4.54	0.54	0.880
		320	1.5	15	13.50	4.44	0.54	0.892
B-H	5	0	13.30	4.27	0.56	...
		160	0.5	30	10.72	3.46	0.59	0.266
		160	0.5	15	10.32	3.43	0.59	0.319
		320	0.5	15	9.82	3.25	0.61	0.360
		320	1.5	15	7.49	2.51	0.67	0.600
B-H	15	0	88.46	22.02	0.40	...
		160	0.5	30	75.10	18.81	0.42	0.157
		160	0.5	15	67.41	17.53	0.41	0.248
		320	0.5	15	65.64	16.82	0.43	0.269
		160	1.5	15	42.20	10.79	0.48	0.567
		320	1.5	15	40.34	10.42	0.48	0.568
A-L	5	0	6.19	2.25	0.67	...
		160	0.5	30	5.88	2.15	0.68	0.121
		160	0.5	15	5.67	2.06	0.69	0.202
		320	0.5	15	5.51	2.03	0.69	0.268
		320	1.5	15	4.97	1.79	0.74	0.475
A-L	15	0	17.54	5.76	0.50	...
		160	0.5	30	15.37	5.13	0.52	0.156
		160	0.5	15	14.90	4.95	0.51	0.190
		320	0.5	15	14.69	4.76	0.53	0.205
		160	1.5	15	12.46	4.10	0.56	0.365
		320	1.5	15	12.24	3.95	0.58	0.381
B-L	5	0	5.72	1.93	0.76	...
		160	0.5	30	5.67	1.91	0.75	0.024
		160	0.5	15	5.64	1.91	0.76	0.038
		320	0.5	15	5.63	1.90	0.75	0.043
		320	1.5	15	5.54	1.84	0.78	0.086
B-L	15	0	14.10	3.94	0.63	...
		160	0.5	30	13.79	3.87	0.63	0.029
		160	0.5	15	13.68	3.84	0.63	0.040
		320	0.5	15	13.60	3.80	0.64	0.048
		160	1.5	15	13.36	3.74	0.65	0.071
		320	1.5	15	13.31	3.73	0.65	0.075

^a See Table I for identification. ^b Fraction of polymer contribution to solution viscosity lost through shear.

Reference to these data showed that, for any given level of polymer concentration or sonic test severity, the polymers' order of shear stability was as follows:

1. B-L (most stable)
2. A-L
3. B-H
4. A-H (least stable)

Comparison of the two polyisobutylene solutions, B-L and B-H, and the two polymethacrylate solutions, A-L and A-H, confirmed that shear lability varied directly with molecular weight.

However, when the four additives were compared without regard to chemical type, shear lability did not vary directly with molecular weight—for example, A-L with a molecular weight of 92,000 was more shear-stable than B-H with a lower molecular weight of 44,000. This lack of correlation was also reflected in comparisons using the relative viscosity data (Table I). Analogous observations led Selby (76) to postulate that polymethacrylates have a lower viscosity average molecular weight in mineral oils than in the solvents normally used for molecular weight determinations and that shear stability might be related to polymer solution properties.

A positive correlation was, in fact, found between the ASTM slope of the unsheared solutions and their shear lability. This is illustrated in Figure 1, where the slope is plotted against fraction lost due to shear for each polymer solution. Curves 1 and 2 for the 1.5-ampere tests on the 5 and 15% solutions showed a nearly linear relationship. Curves 3 and 4 for the 0.5-ampere tests were not linear although the ASTM slope still varied directly with shear lability.

The difference between the appearance of the 1.5-ampere (1 and 2) and the 0.5-ampere test curves (3 and 4) suggested that the severity of the sonic treatment affected the degradation of the polymers. Effects of sample volume variation on shear stability had been previously observed (77). This factor and that of varying oscillator input power were examined by calculating the ratios of viscosity loss at several severity levels for the 15% solutions. Table III shows that the three most stable polymers showed greatest response to an increase in oscillator power input. The two polyisobutylenes tended to show higher response to reduction of the sample volume than the polymethacrylates.

These results indicated that polymer response to change in shear stress intensity might be related to polymer chemical

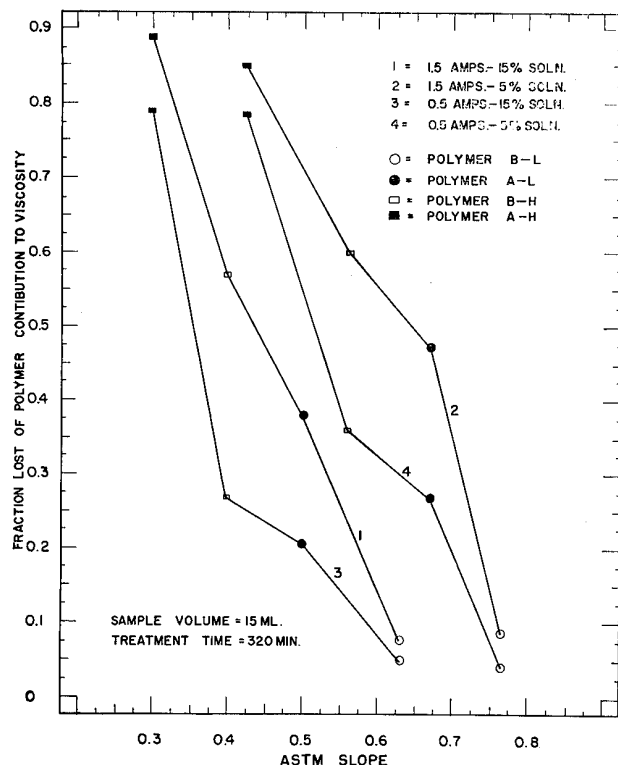


Figure 1. Relation of polymer solution slope to amount of polymer shear at two power levels

type and the intrinsic stability of the polymer. An example of how variation in sonic shear intensity level can affect comparative measurements is provided by the 5% solutions of A-H and B-H (Table II). At a low intensity level (0.5-ampere power, 320 minutes), the B-H loss of polymer contribution to viscosity was less than one half that shown by the A-H (0.360 and 0.786). At the higher intensity level (1.5 amperes, 320 minutes), the B-H loss was nearly three fourths of that shown by A-H (0.600 and 0.852).

Because the ASTM slopes of the unsheared solutions had been implicated as a predictive factor in permanent shear of the polymers, the effect of sonic treatment on this property was further examined. Table IV indicates that the increase of slope (which reflected reduced V-T slope improver power) during shear varied directly with the unsheared solution slope. Loss in this V-T property also consistently paralleled sonic shear lability. Thus, a polymer solution resistant to permanent shear could be expected to show little change in V-T slope under sonic treatment. This behavior had been described by Neunherz (13) for a series of 10W-30 motor oils. Table IV also lists the slope change during shear as a percentage function of the unsheared solution slope. This ratio varied only slightly between the 5 and 15% solutions of the B-L and B-H polymers; however, the A-L and A-H solutions both showed a considerably larger slope increase for the 15% solutions. These results suggested that polymer concentration can influence the degree and type of sonic-induced fragmentation of polymethacrylates.

Another difference in polymer shear effects was observed in comparison of maximum degradation data (Table V). At 5% concentration polymethacrylates A-L and A-H were degraded to similar levels of relative viscosity and ASTM slope. The highly stable polyisobutylene, B-L, was only slightly affected, whereas B-H, despite considerable degradation, re-

Table III. Effect of Varying Sonic Treatment Parameters on Polymer Solution Viscosity

Polymer	Concn., Wt. %	Ratio of Viscosity Loss at 100° F.	
		(1.5 amp.) ^a (0.5 amp.) 15-ml. sample	(15 ml.) ^b (30 ml.) 0.5-amp. power
B-L	15	1.6	1.4
B-H	15	2.1	1.6
A-L	15	1.9	1.2
A-H	15	1.1	1.1

^a Viscosity loss after 320 min. at 1.5 amperes using 15-ml. sample.
Viscosity loss after 320 min. at 0.5 ampere

^b Viscosity loss after 160 min. on 15-ml. sample using input power of 0.5 ampere.
Viscosity loss after 160 min. on 30-ml. sample

Table IV. Effect of Sonic Treatment on ASTM Slope of Polymer Solutions

Polymer	Concn., Wt. %	ASTM Slope, 100–210° F.		
		Unsheared solution	Increase ^a after shear	Increase, %
1. B-L	5	0.76	0.02	2.6
B-L	15	0.63	0.02	3.2
2. A-L	5	0.67	0.07	10.4
A-L	15	0.50	0.08	16.0
3. B-H	5	0.56	0.11	19.6
B-H	15	0.40	0.08	20.0
4. A-H	5	0.42	0.29	69.0
A-H	15	0.30	0.24	80.0

^a Test conditions. 1.5-ampere power, 15-ml. sample, 320 minutes.

Table V. Maximum Sonic Degradation Observed for Polymer Solutions

Polymer	Concn., Wt. %	Solution Property			
		Unsheared		Sheared ^a	
		Rel. viscosity at 100° F.	ASTM slope 100–210° F.	Rel. viscosity at 100° F.	ASTM slope 100–210° F.
A-L	5	1.71	0.67	1.37	0.74
	15	4.85	0.50	3.38	0.58
A-H	5	4.31	0.42	1.49	0.71
	15	25.7	0.30	3.73	0.54
B-L	5	1.58	0.76	1.53	0.78
	15	3.90	0.63	3.68	0.65
B-H	5	3.67	0.56	2.07	0.67
	15	24.4	0.40	11.1	0.48

^a Test conditions. 1.5-ampere power, 15-ml. sample, 320 minutes.

tained a moderate level of thickening and V-T slope improver power. The same is true for the 15% solutions.

Even the least stable solutions showed a small but significant oil thickening and V-T slope improver power compared with the base oil. None of the viscosities or slopes closely approached the corresponding values for the base oil used in this work.

Conclusions

The sonic shear lability of a series of oil solutions of polymethacrylates and polyisobutylenes varied directly with the V-T slope improver power of the unsheared polymers. Sonic degradation of both polymer types reduced their V-T slope improver power to an extent that also varied directly with the unsheared solution slope. The comparative shear lability of the several solutions was affected by the severity level used during the sonic exposure. Prolonged, high severity sonic

treatment did not degrade any of the polymer solutions to the viscosity or V-T slope levels of the base oil used. Polymethacrylates could be degraded further than polyisobutylenes when polymers of equal thickening power were compared.

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1. ORIGINATING ACTIVITY (Corporate author) Rock Island Arsenal Research & Engineering Division Rock Island, Illinois 61201		2 a. REPORT SECURITY CLASSIFICATION Unclassified	
		2 b. GROUP	
3. REPORT TITLE FACTORS AFFECTING SONIC DEGRADATION OF POLYMER SOLUTIONS (U)			
4. DESCRIPTIVE NOTES (Type of report and inclusive dates)			
5. AUTHOR(S) (Last name, first name, initial) LeMar, Ralph L.			
6. REPORT DATE May 1967		7 a. TOTAL NO. OF PAGES 12	7 b. NO. OF REFS 24
8 a. CONTRACT OR GRANT NO.		9 a. ORIGINATOR'S REPORT NUMBER(S) RIA 67-1353	
b. PROJECT NO. DA #1C024401A108		9 b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)	
c. AMS Code 5025.11.802			
d.			
10. AVAILABILITY/LIMITATION NOTICES Distribution of this document is unlimited.			
11. SUPPLEMENTARY NOTES		12. SPONSORING MILITARY ACTIVITY Rock Island Arsenal	
13. ABSTRACT Linear polymers dissolved in mineral oil were examined for their lability to shear degradation utilizing a sonic oscillator. The tests were performed on several polyisobutylenes and polymethacrylates similar to those used to formulate hydraulic fluids with improved viscosity-temperature slope properties. The shear lability of these additives varied directly with their viscosity-temperature slope improver power. Slope improver power also was lost during sonic degradation, its extent depending upon the viscosity-temperature slope of the unsheared solutions. The relative shear lability of the polymer solutions was affected by the severity level employed in the sonic tests. Polymethacrylate solutions could be degraded further than polyisobutylene solutions when additives of equivalent oil thickening power were used; however, none were sheared to the viscosity level of the base oil. (U) (Author			

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Hydraulic Fluids Shear Stability Sonic Oscillators Polymers Polyisobutylene Polymethacrylates						

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FACTORS AFFECTING SONIC DEGRADATION OF POLYMER
SOLUTIONS, by Ralph L. Lemar

RIA Lab. Rep. 67-1353, May 67, 12 p., incl. illus.
tables, (DA Project No. 1C024401A108, AMS Code
5025.11.802) Unclassified report.

Linear polymers dissolved in mineral oil were
examined for their ability to shear degradation
utilizing a sonic oscillator. The tests were
performed on several polyisobutylenes and poly-
methacrylates similar to those used to formulate
hydraulic fluids with improved viscosity-tempera-
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these additives varied directly with their
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temperature slope of the unsheared solutions. The
relative shear stability of the polymer solutions
(Cont.) over

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